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METHOD OF SPECTROGRAPHIC ANALYSIS OF ALUMINIUM

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METHOD OF SPECTROGRAPHIC ANALYSIS OF ALUMINIUM

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METHOD OF SPECTROGRAPHIC ANALYSIS OF ALUMINIUM

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 27 June 1975, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 Considering both the speed and accuracy, recourse is often taken to spectrographic methods for the quick determination of impurities present in aluminium.

0.3 Solid standard method using point to plane spark technique is recommended for spectrographic determination of impurity elements of aluminium of purity 99·0 to 99·8 percent, and intermittent ac arc technique is recommended for determination of purity above 99·8 percent. In addition to plane technique, self pin system may be adopted with suitable excitation parameters.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard recommends spectrographic method for quantitative determination of silicon, copper, manganese, iron and zinc in aluminium.

2. SPARK TECHNIQUE

2.1 General Requirements

2.1.1 *Spectrograph* — Any of the commercial grating or quartz prism instruments that has sufficient resolving power and linear dispersion to separate clearly the analytical lines in the spectrum of the sample in the spectral region 2 300 to 5 000 Å may be used. Of the prism instruments, the large quartz Littrow type, or medium quartz Cornu type may be used. A minimum of 600/mm grating spectrograph with 1·5 m focal length or more may be used.

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*Rules for rounding off numerical values (*revised*).

2.1.2 *Microphotometer* — Any commercial microphotometer providing a standard deviation of ±1 percent transmittance on repeated measurement between 10 and 90 percent transmittances should be used.

2.1.3 *Photographic Plates* — Plates or films as required by the particular instrument should be used.

2.2 Sample Preparation Equipment

2.2.1 *Sample Moulds* — The size, shape and dimension of the sample should be similar to the standard (rod or disc) available for analysis. Sample moulds should be used accordingly. The sample moulds shall be designed to produce homogeneous castings free from any porosity particularly in the region to be sparked and representative of the melt either in the form of rods or in the form of discs. The sample moulds are made of steel or cast iron. In use, the mould surface should be carefully prepared and suitably dressed to minimize the formation of gas pockets in the casting to avoid iron contamination. At the time of casting, the mould temperature should be 200 to 250°C which give satisfactory and sound castings.

2.3 Standard Reference Materials (SRM's)

2.3.1 SRM's used in this method should be in the form of rod or disc covering the range of composition to be determined.

2.4 Preparation of Samples

2.4.1 *Molten Metal* — The sample (either in the form of disc or rod according to the standards available for analysis) is cast in a preheated mould. The mould is preheated by pouring the molten metal into the mould. The preliminary casting thus obtained is, however, discarded. The sample is machined at least 2 mm below the cast surface to a smooth finish for satisfactory sparking.

2.4.2 *Fabricated and Cast Products* — When the metal to be analysed is in wrought or cast form, a representative material sample shall be collected and melted to prepare disc or rod (according to standards available) as described in **2.4.1.** If the sample is in the form of finely divided material, such as turnings or thin sheets, then the grease, or oily substance or any magnetic material should be removed and it should be pressed into lumps for melting and subsequent casting in the form of disc/rod. Melting and casting operation should be done as quickly as possible.

2.4.3 *Other Forms* — If specimens other than cast disc/rod or the forms described in **2.4.1** and **2.4.2** are required to be analyzed, they should be sufficiently massive to prevent undue heating during excitation. Sufficient flat surface may be available for sparking and standards of similar metallurgical conditions should be used. When sample preparation technique described in **2.4.2** cannot be applied, the specimen should be machined to produce a flat surface suitable for sparking. Depth of machining below the original surface of the specimen, location and number of areas to be analyzed should be carefully chosen to provide a representative analysis of the product.

2.5 Excitation and Photography of the Spectra

2.5.1 *Electrode Shape* — Sample electrode with sparking ends machined to give at least 6 mm in diameter with plane end should be used. Just before making each exposure the sparking surface should be carefully turned on a lathe to give plane end.

Counter electrode of high purity graphite or carbon rod of 6 mm diameter tipped at 80° cone should be used.

2.5.2 *Spectrograph* — Large quartz, medium quartz or grating (*see* **2.1.1**) may be used.

2.5.3 *Spark Excitation* — Should be by means of a simple condensed spark circuit, operating on a line voltage of 220-230 volts ac using 1/4 kVA transformer giving an open circuit secondary voltage of 15 kV:

Capacitance	0·005 μF
Added inductance	0·13 mH
Spark gap	4 mm

NOTE — Any other spark excitation source unit and necessary parameters may be used provided similar precision and accuracy are obtained.

2.5.4 *Exposure Conditions*

a) Wavelength range — 2 400 to 3 500 Å approximately (no range specified for medium quartz)

b) Slit width — Prism spectrograph 0·015-0·025 mm
Grating spectrograph 0·01-0·05 mm

c) Slit length — 2 mm

d) Pre-spark period — 10 to 15 seconds

e) Exposure — In general, exposure time is adjusted according to the type of plate used, percentage range of constituents to be analyzed and type of spectrograph used. Normally exposure between 20 to 60 seconds will prove satisfactory

2.5.5 *Photographic Plate/Film* — Kodak B 10; Ilford ordinary N-30 or Eastman Kodak, Spectrum Analysis No. 1. (Exposure time is adjusted according to the type of plate used.)

2.5.6 *Photographic Processing* — The processing of the plates shall be in accordance with the method specified in IS : 6010-1971*.

2.5.7 *Line Pairs for Microphotometer Measurement* — The following lines are compared with Al 2 669·16 Å as internal standard:

Si	2 514·33 Å
Fe	2 719·02 Å
Mn	2 593·73 Å
Cu	3 273·93 Å
Zn	3 345·02 Å

2.5.8 *Analytical Curve* — Spectra of the standards and the samples shall be photographed in duplicate on each plate distributing the spectra all over the plate. The densities of the lines (as recommended under **2.5.7**) shall be measured by microphotometer and converted into line intensities. The average value of log intensity ratio of analytical line to internal standard aluminium line is plotted *versus* log percent of element. A smooth calibration curve shall be drawn for each element and used to determine the unknown percentage.

3. INTERMITTENT ac ARC METHOD (FOR IMPURITIES IN ALUMINIUM ABOVE 99·8 PERCENT PURITY)

3.0 Intermittent ac arc technique method is recommended for quantitative determination of silicon, iron and copper in aluminium above 99·8 percent purity by means of solid standards commercially available in the form of rod or disc.

3.1 General Requirements — as described under **2.1.1** to **2.5.2**.

3.2 Excitation

3.2.1 The sample should be excited by intermittent arc which is adopted from that of K. Pfeilsticker and as described in IS : 2599-1963†.

NOTE — Similar ignited ac arc excitation may be used provided equivalent precision and accuracy are obtained.

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*Recommended practice for photographic processing in spectrochemical analysis.

†Methods of spectrographic analysis of high purity zinc and zinc base alloys for die casting.

3.3 Exposure Conditions

a) Wavelength range — 2 400 to 3 500 Å approximately (no range specified for medium quartz)

b) Slit width — Prism spectrograph 0·01-0·02 mm
Grating spectrograph 0·01-0·05 mm

c) Slit length — 2 mm

d) Arc gap — 3 mm

e) Current — 4-5 A

f) Pre-arcing period — About 10 seconds

g) Exposure — In general, exposure time is adjusted according to the type of plate used, percentage range of constituents to be analyzed and type of spectrograph used. Normally exposure of 20 seconds will prove satisfactory

h) Photographic plate/film — Kodak B 10. Ilford ordinary N-30 or Eastman Kodak Spectrum Analysis No. 1. (Exposure time is also adjusted according to type of plate used)

j) Comparison lines — Si 2 516·12 Å
Cu 3 273·96 Å
Fe 3 020·64 Å
Al 2 567·99 Å

3.4 Analytical Curve

3.4.1 Spectra of the standards and the samples shall be photographed in duplicate on each plate distributing the spectra all over the plate. Develop the exposed plates as described under **2.5.6.** The densities of the lines as recommended under **3.3** shall be measured by microphotometer. The average value of log intensity ratio of analytical line to internal standard aluminium line is plotted *versus* log percent of element. A smooth calibration curve shall be drawn for each element and used to determine the unknown percentage.

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